

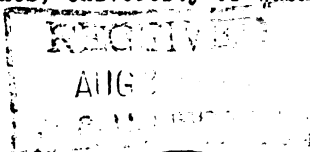
**UNITED STATES
DEPARTMENT OF THE INTERIOR
HAROLD L. ICKES, SECRETARY**

**BUREAU OF MINES
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INFORMATION CIRCULAR

**PORTABLE CABINETS FOR MATERIALS USED
IN MINERAL DETERMINATIONS**

The work upon which this report was based was performed under a cooperative agreement between the Northwest Experiment Station, Bureau of Mines, United States Department of the Interior, and the College of Mines, University of Washington, Seattle, Wash.



BY

KENNETH G. SKINNER

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INFORMATION CIRCULAR

UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

PORTABLE CABINETS FOR MATERIALS USED IN MINERAL DETERMINATIONS^{1/}

By Kenneth G. Skinner^{2/}

INTRODUCTION

Liquids of different specific gravity are invaluable aids in mineral determinations and are essential to methods of sedimentary petrography. The use of such liquids has been more or less restricted to the laboratory because of the difficulty of carrying them into the field. To facilitate field determinations and to aid in the laboratory when it is desirable to move equipment, two portable cabinets were designed and constructed to hold the materials required in the usual mineral determinations.

DESCRIPTION OF CABINETS

A large cabinet was built that held all of the materials needed except the separatory funnels, for which a smaller cabinet was constructed. The larger cabinet was of a size that could be carried by one person for short distances in the field. The following materials are contained in the larger cabinet: Hydrochloric, nitric, and sulfuric acids, ammonium hydroxide, 2 organic solvents, water, 4 bottles to contain washings, 16 liquids of different specific gravity, 9 test tubes, 4 short-stemmed funnels, 4 50-cc. beakers, 3 10-cc. burettes, 1 10-cc. graduate, 1 100-cc. graduate, filter papers, microscope slides and cover glasses, streak plates, and burette clamps.

Figure 1, an isometric drawing of the larger cabinet with the door removed, shows the arrangement of the interior. A sliding door was used because it could be removed and set out of the way, while one on hinges could not. The heavy liquids and filter papers were placed on the bottom, the burettes on racks, and the rest of the materials on the shelf. Three holes, connected by a slot, were made in the top of the cabinet to hold the separatory funnels while they were being used.

Figure 2 is a working drawing (with the door removed) giving dimensions of the cabinet. The location of partitions on the bottom and on the shelf are not shown in the top view. A removable shelf with two prongs of 3/16-inch strap iron may be placed on the end of the cabinet (as indicated by the dashed lines in fig. 2) by slipping the prongs into the slots in the thin board. This shelf

^{1/} The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7127."

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is a convenient place on which to put beakers, etc. The veneer door is re-enforced, top and bottom, by 3/8- by 1-inch wood strips. A drawer pull is employed as a handle on top of the cabinet, and chest handles are used on the ends. The circled letters in figure 2 indicate where the following articles were placed:

A. - Spindle to hold a single burette clamp. The spindle is not shown in figure 1.

B. - Hangers on the back of the cabinet to hold the burettes. When the cabinet is moved, the burettes may be held firmly in place by rubber bands. A detail of the hangers is given in the upper right side of figure 2.

C. - Slotted thin block on the end to hold the removable shelf.

D. - 8-ounce bottle for water.

E. - 8-ounce bottle for an organic solvent such as carbon tetrachloride.

F. - Microscope slides, cover glasses, and streak plates.

G. - Set of minerals for hardness determinations.

H. - Four 4-ounce reagent bottles: hydrochloric, nitric, and sulfuric acids and ammonium hydroxide.

I. - Nine 25-cc. Pyrex test tubes.

J. - Four 50-cc. beakers.

K. - Three-quarter-inch strap-iron support with a spindle at one end and fastened to the shelf at the other, but free to move. The spindle holds a double burette clamp.

L. - A U-shaped strip of 18-gage galvanized iron, slightly bent, to hold a 10-cc. graduate. The 100-cc. graduate is placed horizontally between the end of the cabinet and the block holding the test tubes.

M. - Twenty 4-ounce, dark, ground-glass, stoppered bottles to hold the specific-gravity liquids and washings, are placed in the bottom sections. One bottle of acetone also is placed here. Filter papers are placed in the space between the bottles and the end of the cabinet.

Small screens, a magnet, and a small mortar and pestle may be placed on the shelf.

The smaller cabinet (fig. 3) is used to hold three pear-shaped separatory funnels (two 125-cc. and one 250-cc.) and a centigrade thermometer. When in use, the funnels may be left in the smaller cabinet or placed in the slot in the larger cabinet. The latter method was found to be more satisfactory. The

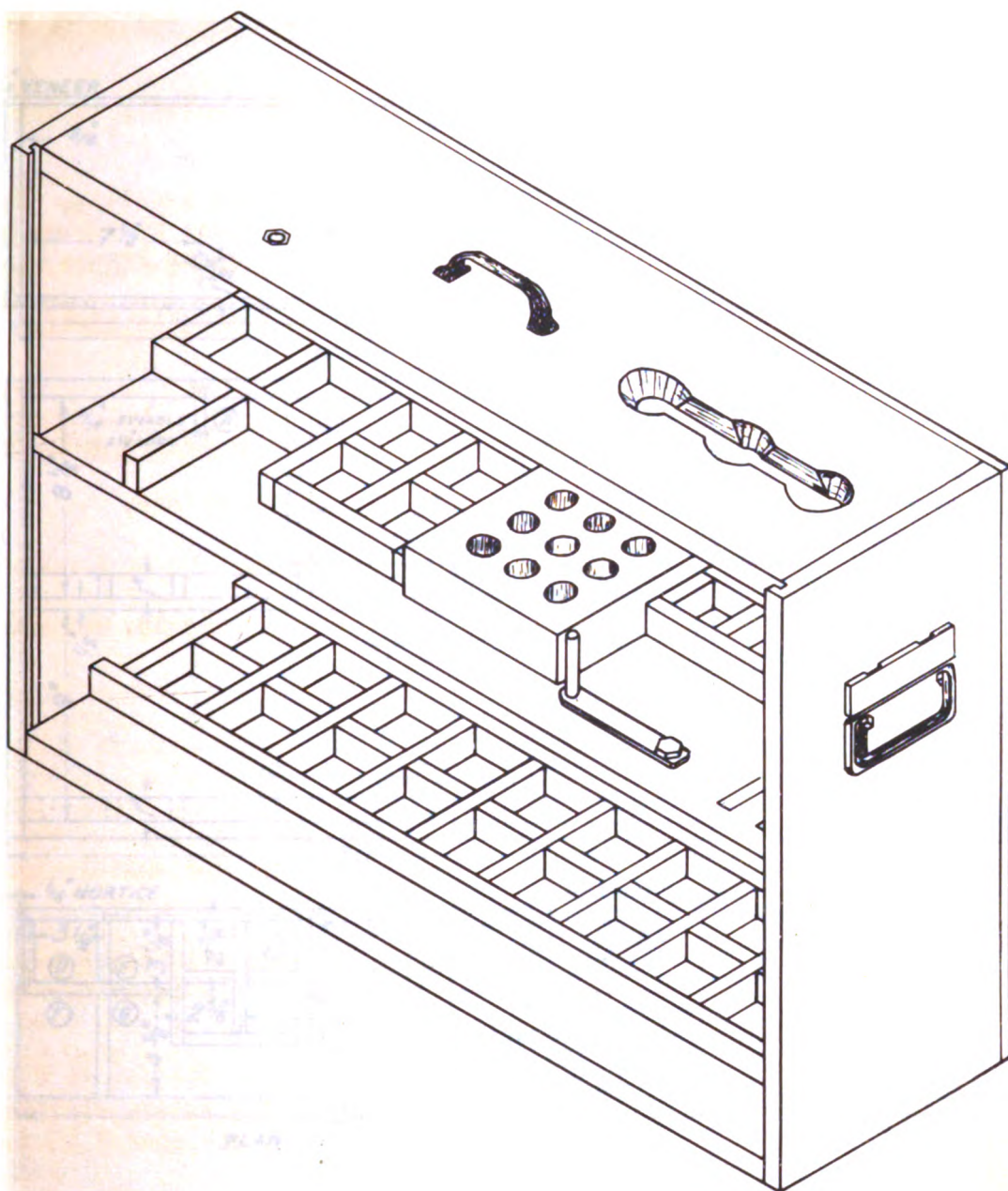


Figure 1.- Isometric drawing of the larger cabinet. Bottles containing liquids of different specific gravities are placed on the bottom, and other supplies and equipment (except separatory funnels) are placed on the shelf.

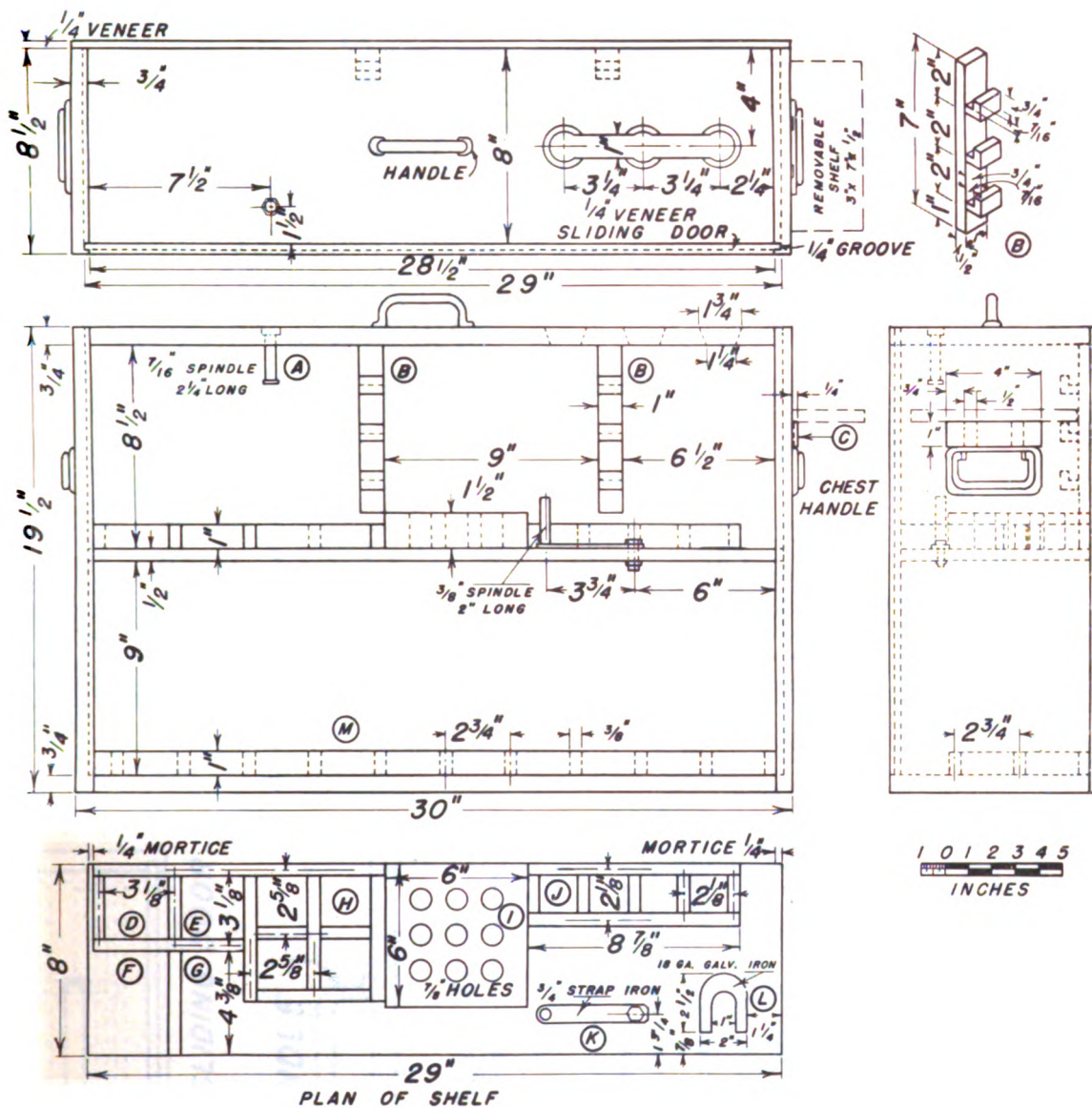


Figure 2.— Working drawing of the larger cabinet.

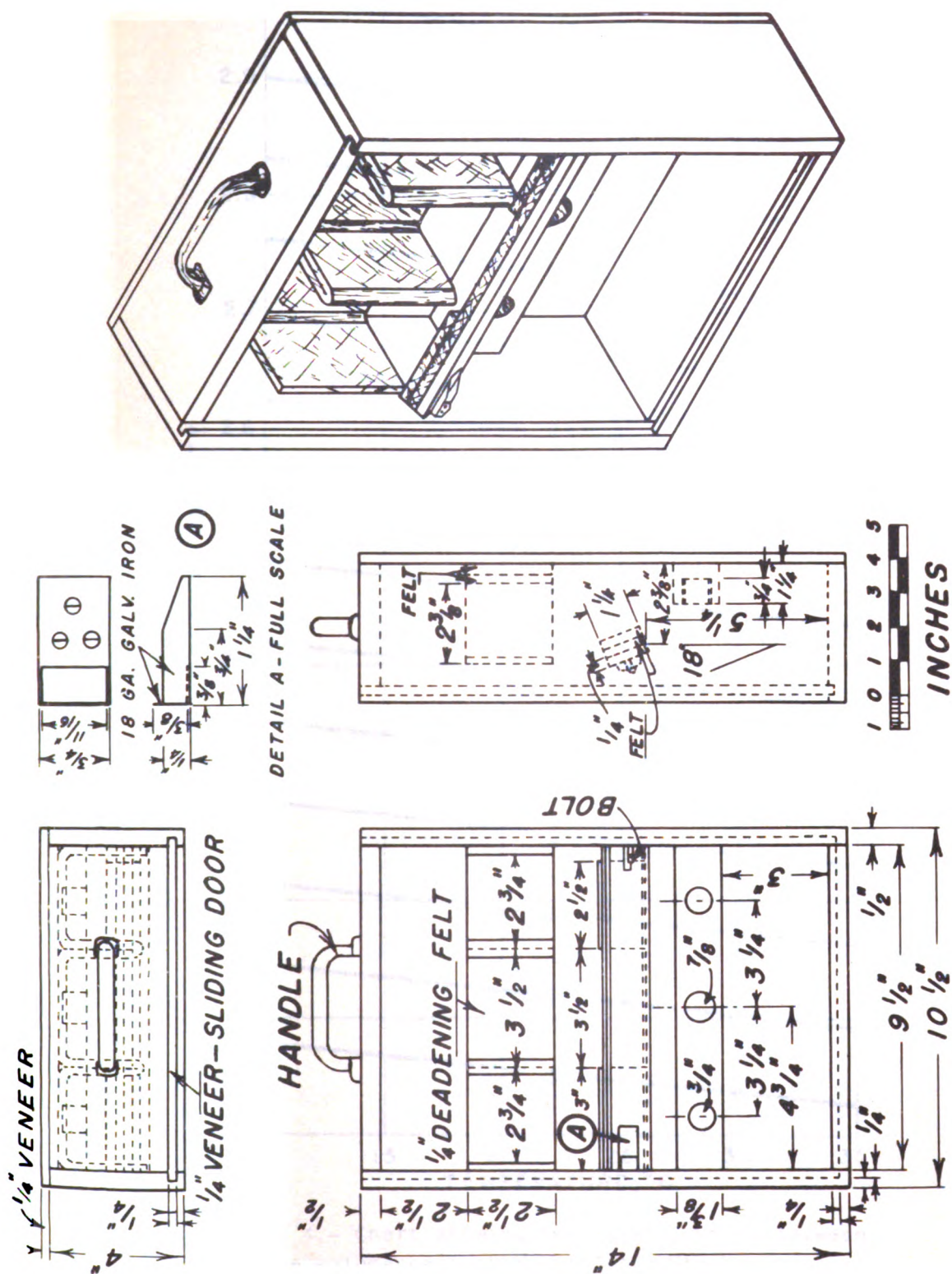


Figure 3.— Working and isometric drawings of the cabinet holding the separatory funnels.

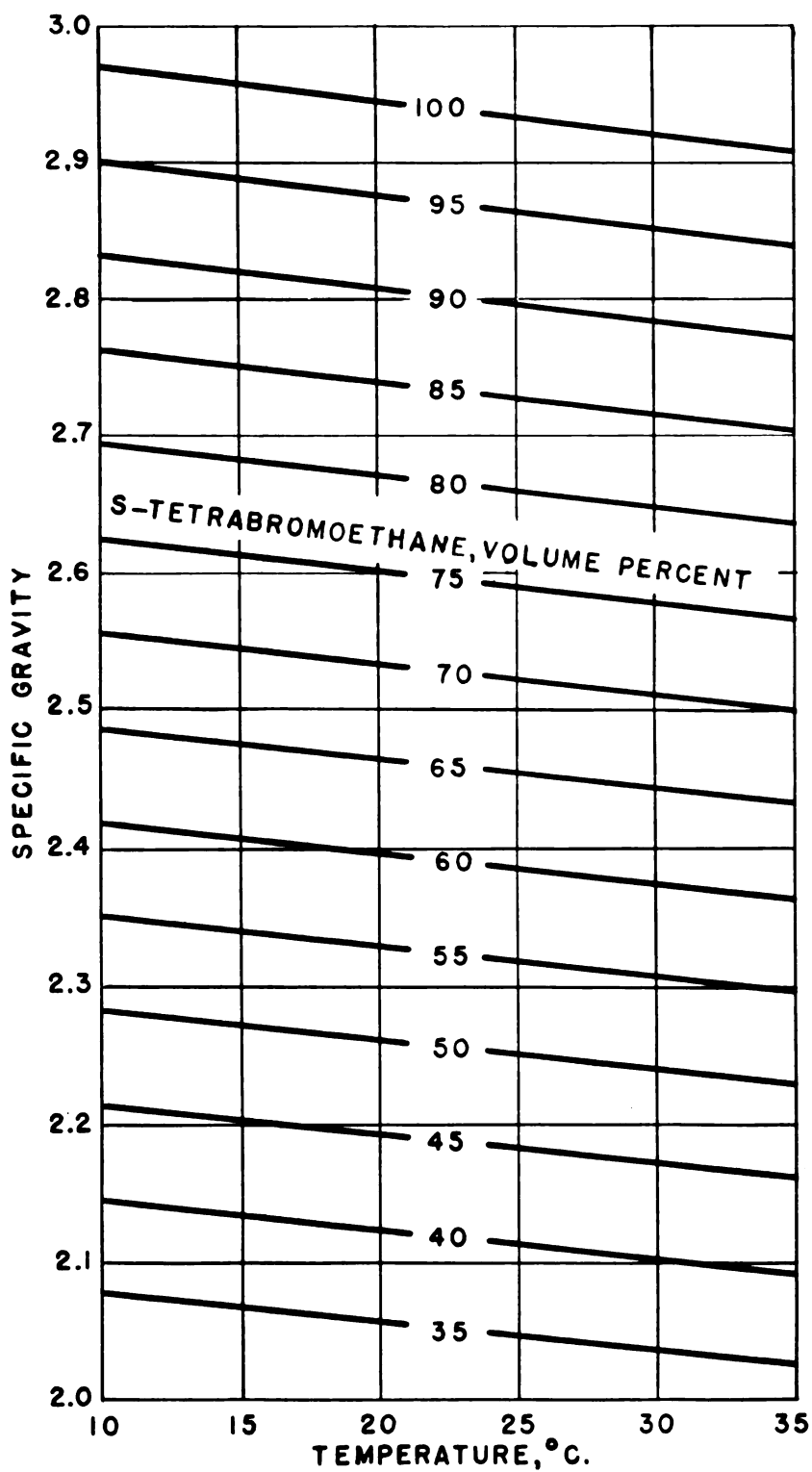


Figure 4.- Chart showing the relationships between the volume percentage of S-tetrabromoethane, specific gravity, and temperature, °C.

veneer door is reenforced, top and bottom, with veneer strips 1 inch wide. The construction of the holder for the felt-backed bar that holds the separatory funnels in place is shown in detail A, figure 3. The funnels are supported by their stop-cocks, which fit into holes in a block fastened to the back of the cabinet.

Heavy Liquids Used

The heavy liquids used in this laboratory ranged in specific gravity from 1.58 to 4.1, which was the range expected for most non-metallic samples. The liquids had the following specific gravity: 1.58, 1.8, 2.0, 2.2, 2.3, 2.4, 2.5, 2.6, 2.7, 2.8, 2.9, 3.0, 3.1, 3.5, 3.8, and 4.1. Bottles to hold washings were put in the blank places in the space allotted for specific-gravity liquids.

Sullivan^{3/} discusses the various liquids used for mineralogical determinations and, following his recommendations, *s*-tetrabromoethane (symmetrical acetylene tetrabromide) and carbon tetrachloride were used as the bases for all of the liquids with specific gravity between 1.58 and 2.9. These chemicals are comparatively cheap and readily obtainable, and it is not difficult to make up the various mixtures. The washings from these determinations should be saved and recovered by fractional distillation.

Thoulet and Clerici solutions were made up according to the usual methods^{4/} and used when the specific gravity was greater than 3.0. Thoulet's solution ($\text{HgI}_2 \cdot 2\text{KI}$) was used for a gravity of 3.0 and of 3.1, and Clerici's solution (thallium malonate and thallium formate) for any between 3.4 and 4.1. These solutions were more viscous than those of lower gravity, and separation of fine minerals (minus 35-mesh) was more difficult, except upon long standing. The water washings from these liquids should be saved so that the expensive chemicals could be recovered by evaporation. Ten cc. of concentrated Clerici solution costs about \$9.

Sullivan^{5/} discusses the effect of temperature on the specific gravity of *s*-tetrabromoethane and of carbon tetrachloride but not for mixtures of these two chemicals. His experimental data showed they could be mixed in all proportions without appreciable change in volume, permitting the calculation of the specific gravity of the resulting solutions.

The relationships among the volume percentages of the commercial *s*-tetrabromoethane and the carbon tetrachloride used in this laboratory, temperature ($^{\circ}\text{C}.$), and specific gravity of the solutions are shown by equation 1 and figure 4.

3/ Sullivan, J. D., Heavy Liquids for Mineralogical Analyses: Bureau of Mines Tech. Paper 381, 1927, 26 pp.

4/ Iddings, Joseph P., Rock-Making Minerals: John Wiley & Sons, New York, 1900, p. 100.

Vassar, H. E., Clerici Solution for Mineral Separation by Gravity: Am. Mineral., vol. 10, 1925, p. 123.

5/ Sullivan, J. D., Work cited in footnote 3, pp. 11-12.

$$(1) \text{ Solution specific gravity} = 0.0136S - 0.00241t + 1.631$$

where $S = \text{s-tetrabromoethane}$

$t = \text{temperature, } ^\circ\text{C.}$

If a liquid of a definite specific gravity is required, the volume percentages may either be calculated from the equation or determined from the chart.

A different equation is obtained (equation 2) if the constants

$$(2) \text{ Solution specific gravity} = 0.0138S - 0.01t (0.19 + 0.003S) + 1.632$$

given in the International Critical Tables are used.

Standardization of the liquids. - The specific gravity of the heavy liquids used for mineral determination were determined by means of pycnometers, and occasionally were checked with a portable Westphal balance. Liquids having a specific gravity greater than 3.0 were so viscous that it was difficult to check their specific gravity with the balance.

Although the specific gravity actually was determined to the third decimal place (thousandths), only values of tenths were considered when mineral separations were being made. Surface tension, oil films, intergrowth of minerals, etc., affect the specific gravity too much to permit use of accurate figures.

Recovery of s-tetrabromoethane. - In this laboratory the mineral fractions obtained from the gravity separations were washed with carbon tetrachloride and then with acetone. It was found necessary to use the acetone to facilitate drying of the mineral fractions. All of the washings used were saved and recovered by fractional distillation, and purification with sodium **thiosulphate** (hypo).^{6/}

The washings were put into a distillation flask and heated to a maximum of 150°C. , and the fraction obtained between 140°C. and 150°C. was retained as s-tetrabromoethane. This fraction contained free bromine, which was removed by adding a water solution of hypo and then separating by means of a separatory funnel. The resultant liquid had a slight amber color and a specific gravity of 2.93° at 22.5°C. , which compared favorably with the original gravity of 2.94° at this temperature.

ACKNOWLEDGMENTS

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^{6/} Procedure established by Anthony Centenero, assistant analytical chemist, University of Washington.

APPENDIX

Bill of Materials

1 piece - 3 x 4-1/2 feet, 1/4-inch veneer, to be cut as follows:

1- 30" x 19-1/4"	1- 9-1/2" x 1-1/4"
1- 29" x 18-3/4"	2- 9-1/2" x 1"
1- 14" x 10-1/2"	1- 4" x 1"
1- 13-3/4" x 10"	

1 piece - 3/4" x 9" (finished) x 8', to be cut as follows:

2- 19-1/2" x 8-1/2"	1- 28-1/2" x 8"
1- 28-1/2" x 8-1/2"	

1 piece - 1/2" x 4" (finished) x 4-1/2', to be cut as follows:

1- 9-1/2" x 3-1/2"	2- 14" x 4"
1- 9-1/2" x 4"	1- 7" x 3"

1 piece - 3/8" x 1" (finished) x 25'

1 piece - 6" x 6" x 1-1/2"

1 piece - 9-1/2" x 1-3/8" x 1-1/2"

1 piece - 6" x 3/4" x 3/4" (6 pieces, 1" x 3/4" x 3/4")

1 piece - 29" x 8" x 1/2"

1 piece - 1/4" deadening felt, 2-1/2" x 36", to be cut as follows:

1- 9-1/2" x 2-1/2"	1- 9" x 1-1/4"
1- 25" x 2-1/2"	1- 3" x 1-1/4"
	1- 2-1/2" x 1-1/4"

1 piece - 18 gage galvanized iron 2" x 3-3/4", to be cut as follows:

1- 2" x 2-1/2"	1- 1-1/4" x 1-1/4"
1- 3/4" x 3/4"	

2 pieces - 3/16-inch strap iron, 1-1/2" long

1 piece - 3/4-inch strap iron, 4-1/2" long

1 - 1/2" x 1" machine bolt and nut

1 - 7/16-inch spindle 2-1/4" long, threaded at one end

1 - 3/8-inch spindle 2" long

1 pair - chest handles

1 pair - large drawer pulls

1 - small cabinet-door bolt

Paint (it is not advisable to use a coal-tar paint soluble in the organic solvent)

Nails

I. C. 7127

Equipment:

- 20 - 4-oz. dark, ground-glass stoppered bottles
- 4 - 4-oz. reagent bottles
 - 1 hydrochloric acid
 - 1 nitric acid
 - 1 sulfuric acid
 - 1 ammonium hydroxide
- 2 - 8-oz. bottles
- 3 - 10-cc. burettes
- 1 - 100-cc. graduate
- 1 - 10-cc. graduate
- 4 - 50-cc. beakers
- 9 - 25-cc. Pyrex test tubes
- 2 - 125-cc. pear-shaped separatory funnels
- 1 - 250-cc. pear-shaped separatory funnel
- 3 - short-stemmed funnels
- 1 - single burette clamp
- 1 - double burette clamp
- Filter papers
- Microscope slides and cover glasses
- Streak plates
- Rubber gloves